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Antiferromagnetic resonance in crystalline $PrFe_3(BO_3)_4$

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Experimental AFMR studies of crystalline $PrFe_3(BO_3)_4$ over a wide frequency range of 10–143 GHz at a temperature of 4.2 K have been carried out. The high-frequency properties of praseodymium ferroborate are well described in terms of a model of a two-sublattice antiferromagnet with an "easy axis" anisotropy. An energy gap of 134.3 ± 0.5 GHz is determined and the magnitude of the effective magnetic anisotropy field is estimated to be 1.9 ± 0.1 kOe. An analysis indicates that the spin-orientational phase transition in this compound is a first order transition. *Published by AIP Publishing.*
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Introduction

Compounds in the extensive isostructural family of rare-earth ferroborates with common formula $RFe_3(BO_3)_4$ (R is a rare-earth ion) have a wide range of magnetic and electrical properties. These multiferroics manifest an entire series of spontaneous and magnetic-field induced phase transitions.[1] The magnetic moments of the $Fe^{3+}(^6S_{5/2})$ ions are ordered antiferromagnetically at temperatures on the order of 30–40 K. The paramagnetic rare-earth ions R^{3+} are effectively magnetized by exchange interactions with the iron subsystem. The effective magnetic anisotropy of the ferroborates with $R = Nd^{3+}, Y^{3+}, Sm^{3+}, Er^{3+}$ is positive ("easy plane" type), while for the crystals with $Tb^{3+}, Dy^{3+}, Pr^{3+}$ it is negative ("easy axis").[1] It should be noted that the magnetoelectric properties of the compounds $RFe_3(BO_3)_4$ have been studied in some detail,[2] while their high-frequency properties have been examined in a limited number of papers. In particular, an antiferromagnetic resonance (AFMR) on the Fe^{3+} ions has been observed in crystalline $GdFe_3(BO_3)_4$,[3] $Nd_{0.75}Dy_{0.25}Fe_3(BO_3)_4$,[4] and $Nd_{0.75}Ho_{0.25}Fe_3(BO_3)_4$,[5] and the results are well described by a simple model of a uniaxial two-sublattice antiferromagnet.[6]

This paper is a study of the single-crystal $PrFe_3(BO_3)_4$ easy-axis antiferromagnet with a trigonal structure belonging to an $R32$ space group. Its magnetic ordering temperature T_N is 30.5 K.[3] The magnetic moments of the Fe^{3+} iron ions in the ordered state are directed along the trigonal axis (the c axis of the crystal).[7] It has been found that a magnetic field **H** directed along the c axis induces a spin-orientation phase transition (the transition field is ~45 kOe at 4.2 K). It is accompanied by discontinuous changes in the magnetization $M(H)$, magnetostriction,[7] and elastic moduli.[8]

Quasioptical studies of $PrFe_3(BO_3)_4$ without a magnetic field indicate an energy gap of $\sim 4.5\,cm^{-1}$ (at $T = 5$ K) of the antiferromagnetic resonance of the Fe^{3+} ion subsystem.[9] The temperature dependence of this energy gap has also been measured. At the same time, no gap associated with the praseodymium subsystem was observed. Optical studies[10] showed that the ground state of the Pr^{3+} ion (the multiplet $^3H_4$) is a singlet level, while the first excited state has an energy of $48\,cm^{-1}$. This indicates that the praseodymium subsystem does not fundamentally change the effective model of a two-sublattice antiferromagnet with an "easy axis" anisotropy that describes the high-frequency properties of this crystal. Its effect can be reduced to an additional contribution to the effective anisotropy field of the iron subsystem by the praseodymium subsystem.

There are few AFMR studies of crystalline $PrFe_3(BO_3)_4$ in the literature. Thus, only preliminary measurements of the frequency-field curve of this substance in an applied field along the c axis have been published.[11] Meanwhile, a detailed study of the high-frequency properties will make it possible to determine the magnitude of the gaps in the spin wave spectrum, evaluate the effective exchange interactions, and obtain additional information on the magnetic structure and nature of the phase transitions. Thus, the goal of this paper is to discover the features of AFMR, evaluate the effective magnetic interactions, and clarify the nature of the magnetic phase transition in single-crystal $PrFe_3(BO_3)_4$.

Characteristics of the sample and technique

Single-crystal praseodymium ferroborate was produced by solution-melt crystallization.[12] The orientation of the crystal axes of $PrFe_3(BO_3)_4$ was determined by x-ray methods. After mechanical processing, the test sample consisted of a thin $3 \times 3 \times 0.1$ mm slab. The trigonal axis was directed perpendicular to the surface of the slab and coincided with the crystallographic c axis. Since defects may form during working of a crystal, the sample was annealed at a high temperature to minimize mechanical stresses.

The field dependences of AFMR spectra in single-crystal $PrFe_3(BO_3)_4$ were studied for frequencies of 10–143 GHz on a system of standard spectrometers at a temperature of 4.2 K. The active element consisted of cylindrical H_{01n}-mode cavities for the corresponding frequency ranges. Only microwave fields with perpendicular polarization were used.

Since single-crystal $PrFe_3(BO_3)_4$ manifests a highly distinct axial symmetry in its magnetic properties, we measured the magnetic resonance for two different orientations of the external magnetic field: along the crystallographic c axis ($\mathbf{H}\|c$) and perpendicular to it ($\mathbf{H}\perp c$). The error in the orientation of the sample was less than 0.1°. Experiments with an inclined magnetic field at a small angle to the c axis of the crystal were also carried out.

Experiment

Figure 1 shows a series of microwave power absorption spectra of single-crystal $PrFe_3(BO_3)_4$ for frequencies ranging from 12 to 143 GHz with a $\mathbf{H}\|c$ orientation of the external magnetic field. Since a phase transition is observed in a sample with this orientation at a field of $H_t = 45.4 \pm 0.2$ KOe, indicated in the figure by a vertical dashed line (a feature in the form of a "step" is observed at this field in the AFMR spectra for almost all the frequencies accessible in this experiment), it is logical to examine the resonance behavior of the crystal separately in fields below and above H_t.

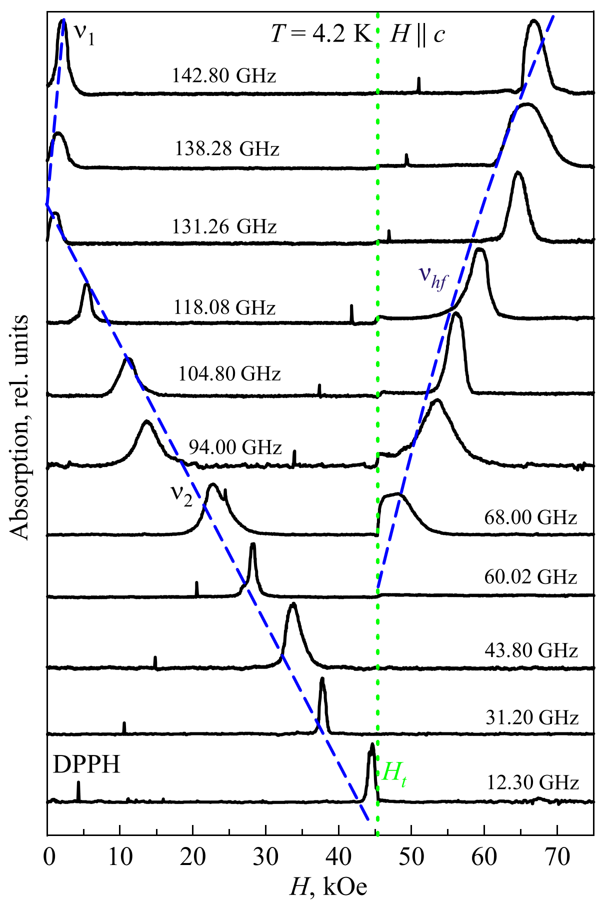

Fig. 1. AFMR absorption spectra in single-crystal $PrFe_3(BO_3)_4$ within a frequency range of 12–143 GHz with $\mathbf{H}\|c$. The dashed lines are schematic illustrations of the variation in the resonance field of the AFMR modes ν_1, ν_2, and ν_{hf} with observation frequency. The vertical dashed line indicates the field $H_t = 45.4$ kOe for the phase transition. The narrow line corresponds to the signal from a standard sample of diphenyl picryl hydrazyl (DPPH).

For $H < H_t$, two linear AFMR modes ν_1 and ν_2 are observed coming from a single gap Δ of magnitude 134.3 $\pm$ 0.05 GHz. The resonance field for mode ν_1 increases with the frequency of the observations, while that for mode ν_2 falls off monotonically. Because of experimental limitations, frequencies in the rising branch of ν_1 were observed only within a small frequency interval of 134.3–143 GHz. On the other hand, the softening of the linear AFMR mode ν_2 can be detected over a side range of fields up to H_t. This mode is not detected at fields above H_t. It should be emphasized that the resonance frequency ν_2 in a field of H_t is still nonzero and equals about 12 GHz.

In magnetic fields $H > H_t$, a new mode ν_{hf} is detected with a resonance frequency that increases nonlinearly with rising field. This branch of the oscillations may be associated with a spin-flop mode of an inverted state of a two-sublattice antiferromagnet with an "easy axis" anisotropy. In terms of the model of Ref. 6, the frequency of the spin-flop mode should be equal to zero during the phase transition and then increases as $H^{1/2}$ as the external field is raised. In the experiment, however, the resonance mode ν_{hf} is observed only at frequencies above 65 GHz and is detected up to 143 GHz. Near H_t, no additional resonance absorption lines were found at frequencies from 10 to 65 GHz and from 65 to 143 GHz. Thus, the experimental data manifest an energy discontinuity between the ν_1-ν_2 modes and ν_{hf}.

Figure 2 shows microwave power absorption spectra in single crystal $PrFe_3(BO_3)_4$ for a field orientation $\mathbf{H}\perp c$. In this case, only one mode $\nu_\perp$ is observed; the change in the resonance field for it is indicated by a dashed curve in the figure. This branch of the oscillations can be attributed to the so-called "quadratic mode" of AFMR in a two-sublattice antiferromagnet with "easy axis" anisotropy when the external magnetic field is oriented perpendicular to the easy axis. The resonance field of the observed $\nu_\perp$ mode increases as the microwave frequency is increased. The $\nu_\perp$ branch has an antiferromagnetic gap with the same magnitude $\Delta = 134.3$ GHz as the ν_1 and ν_2 oscillations for $\mathbf{H}\|c$. No resonance lines were observed below the gap for $\mathbf{H}\perp c$.

Besides the main experiment, we have studied AFMR in magnetic fields with a small angular deviation from the c

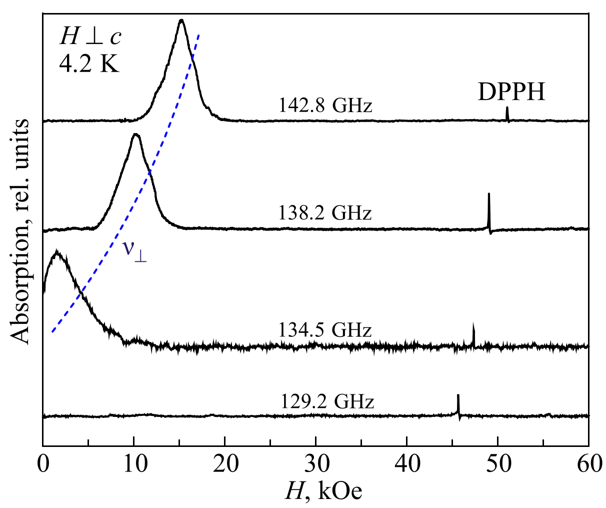

Fig. 2. AFMR absorption spectra in single crystal $PrFe_3(BO_3)_4$ for $\mathbf{H}\perp c$. The dashed curve shows the behavior of the quadratic mode $\nu_\perp$ of the magnetic resonance.

axis. As a comparison, Fig. 3 shows two AFMR absorption spectra in single crystal $PrFe_3(BO_3)_4$ at a frequency of 104.8 GHz in fields of 30–65 kOe with two orientations: (1) **H** strictly coincident with the direction of the *c* axis of the crystal (0°; $\mathbf{H}||c$) and (2) with a deviation from the *c* axis by ~3°. It is clear that for strict $\mathbf{H}||c$ orientation, when $H > H_t$ only a single absorption line ν_{hf} is observed which is detected in a resonant field of about 56 kOe. Besides this line, an anomaly in the form of a "step" in the field H_t (indicated by an arrow in Fig. 3) shows up clearly in the AFMR spectrum. In an inclined field, besides the ν_{hf} line, for $H > H_t$ an additional absorption line (indicated as ν_{SO} in Fig. 3) is detected. We note that as the observation frequency is lowered, the ν_{SO} and ν_{hf} absorption lines move toward one another until they merge into a single resonance peak. Here the frequency-field curve for $PrFe_3(BO_3)_4$ in an inclined field (~3°) has a minimum with coordinates on the order of 51 kOe and 89 GHz (see the inset to Fig. 3).

Discussion

Based on our experimental data taken over a wide range of frequencies and magnetic fields for two orientations of the field (along and perpendicular to the *c* axis), we have constructed the frequency-field curve for AFMR in single-crystal $PrFe_3(BO_3)_4$ shown in Fig. 4. It should be noted that our measurements for $\mathbf{H}||c$ are consistent with previously published data,[11] although we have studied the frequency-field curve in much more detail, especially near the phase transition fields and above them.

We describe the high-frequency properties of $PrFe_3(BO_3)_4$ using a simple model of a collinear two-sublattice antiferromagnet with an "easy axis" anisotropy with the easy axis directed along the c axis of the crystal.[6] For $\mathbf{H}||c$ and $H < H_t$, the two AFMR linear modes ν_1 and ν_2 are described by the following expression:

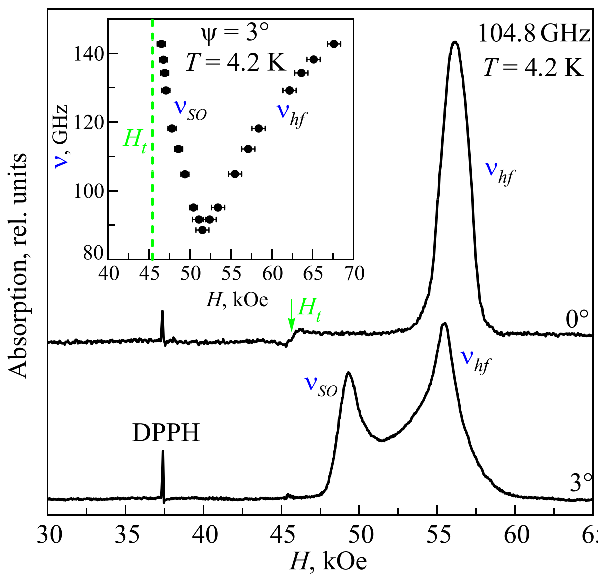

Fig. 3. AFMR absorption spectra in single-crystal $PrFe_3(BO_3)_4$ at a frequency of 143 GHz in an external magnetic field aligned strictly parallel to the *c* axis (0°) and at a slight angle to it (~3°). The inset shows a fragment of the frequency-field curve for AFMR in magnetic fields oriented at angles of ~3° to the *c* axis. The error in determining the resonance field of the AFMR absorption line is indicated by the horizontal error bars. The field H_t for the phase transition is indicated by the dashed line.

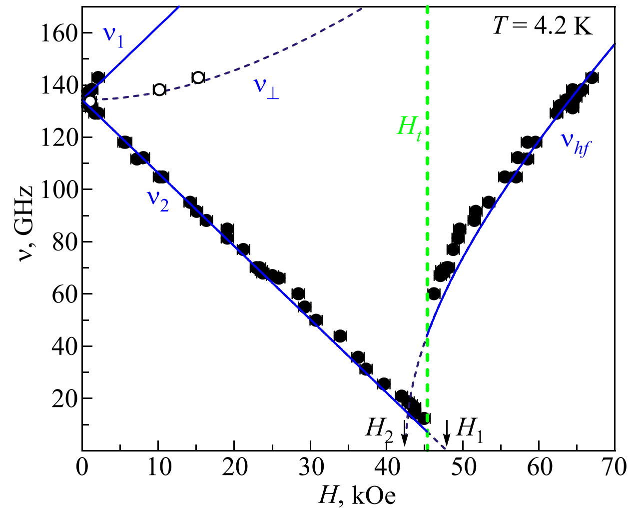

Fig. 4. Frequency-field curve for the AFMR spectrum in single crystal $PrFe_3(BO_3)_4$ for $\mathbf{H}||c$ (●) and $\mathbf{H}\perp c$ (○). The smooth ($\mathbf{H}||c$) and dashed ($\mathbf{H}\perp c$) curves were calculated using the model of a uniaxial two-sublattice antiferromagnet. The vertical dashed line indicates the field of the phase transition $H_t = 45.4$ kOe. The arrows indicate the lability fields H_1 and H_2.

$$\left(\frac{\nu_{1,2}}{\gamma}\right)^2 = \left(\frac{\Delta}{\gamma} \pm H\right)^2, \tag{1}$$

where $\gamma = g\mu_B/h$ is the gyromagnetic ratio (g is the effective g-factor of the Fe^{3+} ion, μ_B is the Bohr magneton, and h is the Planck constant). The "+" sign corresponds to the rising mode ν_1 and the "−" sign to the falling mode ν_2. The best agreement with the experiment is obtained for the following values of the two independent parameters: $\Delta = (134.3 \pm 0.5)$ΓΓ GHz and $\gamma = (2.799 \pm 0.025)$ΓΓ GHz/kOe. The curves for these values are shown as the continuous straight lines in Fig. 4. The estimate of Δ found by this analysis is fully consistent with the experimentally determined value of the AFMR gap in zero magnetic field. In addition, when this value is rescaled in units of inverse centimeters ($4.48\,cm^{-1}$) it is almost exactly the same as the energy gap for the antiferromagnetic resonance ($4.5\,cm^{-1}$) found in quasioptical studies at $T = 5$ K.[8] This value of the parameter γ yields an effective g-factor of $g = 2.00 \pm 0.01$, which fully confirms the expected pure spin state of the Fe^{3+} ions ($^6S_{5/2}$). The magnitude of the AFMR gap can be expressed in terms of the effective exchange field H_e and the magnetic anisotropy H_a of an antiferromagnet as $\Delta/\gamma = (2H_eH_a)^{1/2} = 48.0$ kOe. Using a value of $H_e \approx 600$ kOe for the exchange field of praseodymium ferroborate,[10] the effective magnetic anisotropy field can be estimated to be $H_a = (1.9 \pm 0.1)$ kOe. Finally, it should be noted that for these values of the parameters Δ and γ, the frequency of the AFMR mode goes to zero in a magnetic field of $H_1 = \Delta/\gamma = 48.0$ kOe (see Fig. 4), a value significantly higher than the experimentally determined phase transition field $Ht = 45.4$ kOe.

For $H > H_t$ the field dependence of the resonance absorption line ν_{hf} (the so-called spin-flop mode[6]) is given by

$$\left(\frac{\nu_{hf}}{\gamma}\right)^2 = H^2 - H_2^2, \tag{2}$$

where H_2 is the lability field of the mode at which its resonance frequency goes to zero. The best agreement with the experimental data is obtained for the following values of the two independent parameters: $H_2 = (42.5\pm0.5)$ kOe and $\gamma = (2.80\pm0.05)$ GHz/kOe. The calculated curve is shown as a smooth curve in Fig. 4. The estimate of γ obtained by this analysis is consistent with the value obtained previously for $H < H_t$, while the effective field H_2 is considerably below the experimentally measured transition field H_t. On the other hand, as shown above, the AFMR frequency $\nu_2(H)$ goes to zero for a field H_1 considerably higher than H_t. Thus, the transition field lies within a fairly wide interval between the two lability fields H_1 and H_2. It should be noted that the fixed values of the effective parameters H_e and H_a estimated above from the AFMR gap should predetermine the values of the lability fields H_1 and H_2, respectively, as 47.98 and 47.83 kOe in terms of the model for a two-sublattice antiferromagnet with an "easy axis" anisotropy[6] and the interval between them (of order 150 Oe), which does not agree with experiment. We attribute this large difference between the lability fields H_1 and H_2 [in crystalline $PrFe_3(BO_3)_4$ $H_2 - H_1 \approx 5.5$ kOe] to the magnetizing effect of the praseodymium subsystem on the Fe^{3+} subsystem, which is manifested in the various values of the effective anisotropy field in different magnetic states.

For a perpendicular orientation of the external magnetic field, the model of a two-sublattice antiferromagnet with "easy axis" anisotropy predicts an AFMR mode that is quadratic in the field, with a field dependence of the form

$$\left(\frac{\nu_\perp}{\gamma}\right)^2 = \left(\frac{\Delta}{\gamma}\right)^2 + H^2. \qquad (3)$$

Calculations show that in this case the parameters Δ and γ are identical to the results of the preceding analysis. The corresponding curve is indicated by the dashed curve in Fig. 4. The calculation is in satisfactory agreement with the experimental data.

Thus, the complete AFMR frequency-field curve for single-crystal $PrFe_3(BO_3)_4$ ($\mathbf{H}||c$; $\mathbf{H}\perp c$) can be described satisfactorily in terms of this simple model of a two-sublattice "easy axis" antiferromagnet. We note that in this model the phase conversion of a collinear magnetic state into the reverse (a spin-flop transition) is usually a first-order phase transition.[6]

It is known that when the magnetic field deviates by an angle Ψ larger than some critical value Ψ_{cr} from the easy axis, the phase transition in a uniaxial antiferromagnet no longer occurs discontinuously (typical of a first-order transition) but proceeds via a smooth reorientation of the magnetic moments. The critical angle Ψ_{cr} is determined by the ratio $H_a/H_{e.}$[13] The values of H_a and H_e for crystalline $PrFe_3(BO_3)_4$ yield an estimate of $\Psi_{cr} = H_a/H_e \approx 0.2^\circ$.

For a small $\Psi > \Psi_{cr}$B, an additional absorption line ν_{SO} (which is absent when the magnetic field is strictly oriented along the "easy axis" of the crystal) shows up near the phase transition field in the AFMR spectrum. We assume that the ν_{SO} mode we have observed joins the branches of the AFMR before and after the phase transition in a continuous manner (see Fig. 3). A branch of this kind, which joins the rising linear mode $\nu_1(H < H_t)$ and the spin-flop mode of the inverted state ($H > H_t$) and ensures continuity of the frequency-field curve, usually shows up as an "orientational" resonance.[6]

It can be seen that the AFMR frequency-field curve for $\Psi \approx 3^\circ$ (see the inset to Fig. 3) obtained here is typical of easy-axis antiferromagnets in an inclined field.[6]

Our studies of AFMR indicate that a first-order spin-orientational phase transition (a spin-flop transition) is observed for $\mathbf{H}||c$ in single-crystal $PrFe_3(BO_3)_4$. The main arguments in favor of a first-order magnetic phase transition are the following: (1) a significant frequency gap between the branches ν_1 and ν_{hf} near the phase transition when the field is oriented strictly along the "easy axis"; (2) observation of an "orientational" mode (resonance) when the external field deviates from the "easy axis" by more than a critical angle Ψ_{cr}. In addition, the observed energy gap for excitation of the high field mode ν_{hf}, which is detected only at frequencies above 65 GHz ($H > H_t$), as well as the fact that the oscillation frequency ν_2 does not reach zero at H_t and the corresponding mode vanishes at higher fields, may indicate that the phase transition takes place discontinuously.

On the other hand, theoretical studies indicate[14] that the spin-orientational phase transition in $PrFe_3(BO_3)_4$ from the antiferromagnetic phase into the inverse is a second-order transition and only appears outwardly to be a spin-flop transition. This result is based on the assumption that a weak additional component of the magnetic anisotropy of a higher order is present in the crystal along with the dominant easy-axis anisotropy. Although the constant for this anisotropy is three orders of magnitude smaller than for the easy-axis anisotropy, its existence should mean that this will be a second order transition. A final clarification of the order of this magnetic phase transition in crystalline $PrFe_3(BO_3)_4$ will require further study.

In conclusion, we have made a detailed experimental study of the AFMR in single-crystal $PrFe_3(BO_3)_4$ over a wide range of frequencies, 10–143 GHz, for $\mathbf{H}||c$ and $\mathbf{H}\perp c$ orientations of the external magnetic field at a temperature of 4.2 K. It has been shown that the high-frequency properties of praseodymium ferroborate are described qualitatively by a simple model of a two-sublattice antiferromagnet with an easy-axis anisotropy parallel to the crystallographic c axis. The AFMR frequency-field curve for single-crystal $PrFe_3(BO_3)_4$ for a temperature of 4.2 K has been obtained. An energy gap of $\Delta = (134.3\pm0.5)\Gamma\Gamma$ GHz in the spin wave spectrum of the antiferromagnet and an effective g-factor for the Fe^{3+} ion of $g = 2.00\pm0.01$ have been obtained, and the effective anisotropy field has been estimated to be $H_a = (1.9\pm0.1)$ kOe. The resonance properties of the crystal within the frequency range studied here are determined exclusively by the iron subsystem. These high-frequency studies of $PrFe_3(BO_3)_4$ confirm that when the magnetic field $\mathbf{H}$ is oriented along the c axis, the induced spin-orientational transition is a *first-order* phase transition.

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